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USE OF AN ETHYLENE/ACRYLATE HMA OF 2-ETHYLHEXYL FOR THE PRODUCTION OF ARTICLES SUCH AS PACKAGING

[Utilisation d'un HMA Ethylene/Acrylate de 2-Ethylhexyle pour la Fabrication d'Articles tells que des Emballages}

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DESCRIPTION

This invention relates to the use of a hot melt adhesive (HMA) on the base of a copolymer of ethylene and an alkyl (meth)acrylate, in particular, 2-ethylhexyl acrylate for the production of articles as well as a packaging comprising such a hot melt adhesive.

French Patent Application PCT/FR98/02409, filed by applicant, already described adhesives of the HMA type comprising heavy (meth)acrylates of the 2-ethylhexyl acrylate type, providing better performance at low temperature when compared to butyl or ethyl acrylate applicable at low temperature, for example, between 130 and 150°C.

Applicant, as a matter of fact, discovered that alkyls having at least 5 carbon atoms endow HMA with good properties at low temperature.

The invention intends to use hot melt adhesives (Hot Melt Adhesive or HMA) that present improved practical application performances such as the better hold of the glue seam when exposed to cold, ease of implementation with a short exposure time and a short setting time, great resistance to heat, and

[1] Numbers in the margin indicate pagination in the foreign text.

good thermal stability with good adhesion, both in response to cold and hot.

The hot melt adhesives are solid thermoplastic materials at ambient temperature and, as a result of heating, they turn into viscous liquids.

The HMA adhesives are deposited hot in the form of a viscous liquid on a substrate such as, for example, a strip made of paper, cardboard or polyamide, a polyester substrate, or made of wood or polyolefin upon which applies another substrate in such a way that the HMA will be arranged in sandwich fashion between the two substrates. By cooling down to ambient temperature, we can solidify the two substrates, one upon the other, as a result of the HMA.

The HMA used in the invention can be used in various applications, especially in fields such as:

- packaging of deep-frozen products intended to be stored in a freezer locker requiring an adhesive that will resist low temperatures;

- packaging of heavy products that require a high-performance adhesive;

- glues for wood, book binding, automobiles, buildings, among others.

In the field of packaging, these HMA are applied upon /2

the flaps of the latter after being put into the form of the packaged plate, thus keeping said packaging in shape according to the invention. The packaging items concerned here are made of cardboard or the like.

The invention relates to the use of a hot melt adhesive (HMA) for the production of articles comprising:

- 20 to 50% by weight of 2-ethylhexyl ethylene/acrylate copolymer (A) having an MFI of at least 5, advantageously between 5 and 100, and comprising 25 to 45% by weight of 2-ethylhexyl acrylate;
- 35 to 55% by weight of a tackifying resin or a mixture of tackifying resins;
- 10 to 30% by weight of a wax;
- possibly charges, stabilizers and/or antioxidants;

and having an open time of between 10 and 20 seconds, advantageously between 12 and 15 seconds, and a setting time between 1 and 10 seconds, advantageously between 5 and 7 seconds.

The invention also relates to a packaging comprising a hot melt adhesive (HMA) comprising:

- 20 to 50% by weight of 2-ethylhexyl ethylene/acrylate copolymer (A) having an MFI of at least 5, advantageously between 5 and 100, and comprising 25 to 45% by weight of 2-ethylhexyl acrylate;

- 35 to 55% by weight of a tackifying resin or a mixture of tackifying resins;
- 10 to 30% by weight of a wax;
- possibly charges, stabilizers and/or antioxidants;

and having an open time of between 10 and 20 seconds, advantageously between 12 and 15 seconds, and a setting time between 1 and 10 seconds, advantageously between 5 and 7 seconds.

In the following, reference will be made to different tests or methods of measurement that make it possible to demonstrate the properties of the HMA used in the invention.

Trouble point:

To determine the trouble point: We heat the HMA to 175°C and we deposit a drop of that HMA on the bulb of an ASTM thermometer. The temperature at which trouble occurs during cooling constitutes the trouble point. When the trouble /3 point is at less than about 60°C, that indicates good compatibility between the constituents of the hot melt adhesive.

BROOKFIELD viscosity:

This viscosity is measured at 170°C with a 27 needle according to the ASTM D3236 standard.

SAFT or PAFT:

The SAFT test (or Shear Adhesion Failure Temperature) measures the capacity of a hot melt adhesive to resist a static

force of 500 g while shearing due to the effect of a regular temperature rise by 0.4°C/min.

The PAFT test (or Peel Adhesion Failure Temperature) measures the capacity of a hot melt adhesive to resist a static force of 100 g while peeling due to the effect of a regular temperature rise of 0.4°C/min.

In both cases, we note the temperature at which we observe the rupture of the glue seam.

Open time and setting time:

The open time and the setting time are determined on the OLINGER instrument. The test involves depositing a streak of hot melt adhesive of 0.1 g with the help of a nozzle on a cardboard plate measuring 50 mm by 65 mm. Another cardboard plate with the same dimension is applied upon the glue seam with a pressure of 1 kg/cm^2. The two cardboard plates are then glued upon each other for a defined period of time under a certain pressure.

The setting time is defined as the time at the end of which one gets at least 80% fiber separation after the separation of the two cardboard plates.

The open time is defined as the period during which the streak of glue no longer makes it possible to get a sufficient adhesion between the two cardboard plates, thus characterizing the absence of fiber separation.

Adhesion at low temperature:

Measuring the adhesion at low temperature involves depositing a streak of hot melt adhesive at a temperature of 170°C on a cardboard plate with dimensions of 50 by 65 mm and then, immediately afterward, manually applying a cardboard plate having the same dimension upon the glue seam. The assembly, thus made, is allowed to rest for 24 hours at 23°C and is then placed in a climate-controlled container between -20 and -50°C for 24 hours with a relative humidity of 50%. One then determines the type of rupture as one proceeds to the /4 separation of the two cardboard plates. One then speaks in terms of the fiber separation or fiber non-separation of the cardboard.

Ring ball temperature:

A steel ball with a specific mass is placed upon a sample of adhesive contained in a metallic ring with specified dimensions. The apparatus is heated at a constant defined speed. The softening point is the temperature at which the sample becomes sufficiently soft to permit the ball to pass through the ring over a specified distance.

The tackifying resins have a natural or synthetic origin. The suitable tackifying resins, for example, are colophony, colophony esters, hydrogenated colophony, the polyterpenes and derivatives, the aromatic or aliphatic petroleum resins, the

hydrogenated cyclic resins. These resins typically have a ring ball softening temperature between 25 and 180°C and preferably between 50 and 135°C. One might also mention here the dicyclopentadienes.

The adhesives, used in the invention, contain waxes that make it possible to adjust the fluidity, the open time and the setting time. The waxes can be recovered during the refining of the petroleum fractions. These, for example, are waxes that essentially consist of paraffinic hydrocarbons and that contain sufficient quantities of ramified, cyclic and aromatic hydrocarbons to be much less crystalline than the paraffins. One can also use synthetic waxes.

Advantageously, the waxes comprised in the glues used in the invention have a melting temperature of more than 50°C and preferably between 60 and 70°C.

The HMA, used in the invention, can contain charges. As an example of charges, we might mention silica, alumina, glass, glass balls, calcium carbonates, fibers and metallic hydroxides. These charges must not reduce the tack nor the mechanical properties of the glue after its application.

It is recommended that one add stabilizers such as antioxidants.

The hot melt adhesives, used in the invention, are prepared by mixture in the melted state at temperatures of between 130

and 200°C until one gets a homogeneous mixture. The mixing time can be on the order of between 30 minutes and 3 hours. One can use the usual thermoplastic devices such as extruders, cylinders, Banbury or Brabender mixers or screw mixers. /5

EXAMPLES:

We used the following products:

Lotryl 40EH21: 2-ethylhexyl ethylene/acrylate copolymer with a 2-ethylhexyl content of 40% and an MFI of 21.

Lotryl 30EH65: 2-ethylhexyl ethylene/acrylate copolymer with a 2-ethylhexyl content of 30% and an MFI of 65.

Lotryl 35BA40: butyl ethylene/acrylate copolymer with a butyl acrylate content of 35% and an MFI of 40.

Evatane 28-40: vinyl ethylene/acrylate copolymer with a vinyl acetate content of 28% and an MFI of 40.

Escorez 5600: aliphatic/aromatic tackifying hydrocarbon resin by EXXON.

Sasol paraflint H2: a wax sold commercially by SCHUMAN SASOL.

Irganox 1010: a phenolic antioxidant by CIBA SPECIALITY CHEMICALS.

Table 1 shows various compositions of HMA called SB22 (HMA used in the invention), SB32 (HMA used in the invention), SB42 (comparative 1) and SB52 (comparative 2).

TABLE 1

COMPOSITION	SB22	SB32	SB42	SB52
Lotryl 40EH21	30			
Lotryl 30EH65		30		
Evatane 28-40			30	
Lotryl 35BA40				30
Escorez 5600	55	55	55	55
Sasol paraflint H2	15	15	15	15
Irganox 1010	0.2	0.2	0.2	0.2

Table 2 shows the properties of these HMA. We recall that there is fiber separation when the fibers of the cardboard are torn away at the moment of desolidification of two cardboard panels that are glued upon each other. Fiber separation thus characterizes a strong adhesion of the two panels upon each /6 other. There is glazing when the desolidification of the two cardboard panels, glued upon each other, takes place without fiber separation. Glazing characterizes a lesser adhesion when compared to the previous one.

TABLE 2

CHARACTERISTICS	SB22	SB32	SB42	SB52
Brookfield viscosity 170°C mPas	6900	2930	3200	3720
Softening point, °C	106	101	105	105
Trouble point, °C	97*	92	93	94
Open time, secs.	15	12 to 13	11 to 12	11 to 12
Setting time, secs.	7	5 to 6	7	8 to 9
SAFT, °C	87	84	82	81
PAFT, °C	82	76	72	74
Hold at low temperature				
- 20°C	fiber separation	fiber separation	slight fiber separation	fiber separation
- 30°C	fiber separation	glazing	glazing	fiber separation
- 40°C	fiber separation	glazing	glazing	slight fiber separation
- 50°C	fiber separation	glazing	glazing	slight fiber separation

We find that the compositions of the bonding agents SB22 and SB32, comprising 2-ethylhexyl acrylate, yield better adhesion results than SB42 (comparative 1) at -20°C. We also find that SB22 yields better adhesion results than SB52

(comparative 2) at -40°C and -50°C.

It thus appears that at very low temperatures (below -20°C), SB22 permits strong and invariable adhesion. This adhesion is better at -40°C and -50°C than the one obtained with SB52 (comparative 2) on a base of butyl acrylate (EBA) and, at any rate, better than that obtained with SB42 (comparative 1) on a base of vinyl acetate (EVA). It also appears that SB22, comprising 40% 2-ethylhexyl acrylate, yields better adhesion starting at -30°C, and that SB32, comprising 30% 2-ethylhexyl acrylate, yields better adhesion starting at -50°C.

On the other hand, it appears that HMA SB22 benefits from a better setting time than HMA SB42 at a low EVA and that HMA SB32 benefits from a better setting time than the HMA SB42 or than HMA SB52. We also find that the open times of HMA SB22 and /7 SB32 are essentially longer than those of HMA SB42 and SB52.

These examples make it possible to evidence the superiority of HMA comprising 2-ethylhexyl acrylate when compared to HMA comprising butyl acrylate or vinyl acetate at temperatures lower than -20°C.

CLAIMS /8

1. Use for the production of articles of a hot melt adhesive (HMA), comprising:

- 20 to 50% by weight of 2-ethylhexyl ethylene/acrylate copolymer (A) having an MFI of at least 5,

advantageously between 5 and 100, and comprising 25 to 45% by weight of 2-ethylhexyl acrylate;

- 35 to 55% by weight of a tackifying resin or a mixture of tackifying resins;
- 10 to 30% by weight of a wax;
- possibly charges, stabilizers and/or antioxidants.

2. Use for the production of articles of a hot melt adhesive (HMA) according to Claim 1, said HMA having an open time of between 10 and 20 seconds, advantageously between 12 and 15 seconds, and a setting time of between 1 and 10 seconds, advantageously between 5 and 7 seconds.

3. Packaging comprising a hot melt adhesive (HMA), comprising the following:

- 20 to 50% by weight of 2-ethylhexyl ethylene/acrylate copolymer (A) having an MFI of at least 5, advantageously between 5 and 100, and comprising 25 to 45% by weight of 2-ethylhexyl acrylate;
- 35 to 55% by weight of a tackifying resin or a mixture of tackifying resins;
- 10 to 30% by weight of a wax;
- possibly charges, stabilizers and/or antioxidants.

4. Packaging comprising a hot melt adhesive (HMA) according to Claim 3, said HMA having an open time of between 10 and 20 seconds, advantageously between 12 and 15 seconds, and a setting

time of between 1 and 10 seconds, advantageously between 5 and 7 seconds.